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WATER-DISPERSIBLE GRANULAR AGROCHEMICAL COMPOSITION
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[Claims] /2*

[Claim 1] A water-dispersible granular agrochemical composition comprising one or more kinds of agrochemical active ingredients, surface-active agent 1 below, and surface-active agent 2 below:

(1) one or more kinds of surface-active agents selected from lignosulfonic acid, naphthalenesulfonic acid formalin condensate, maleic acid-diisobutylene copolymer, maleic acid-isobutylene copolymer, and their alkali metal salts, alkaline earth metal salts, ammonium salts, and amine salts and

(2) one or more kinds of surface-active agents selected from alkylnaphthalenesulfonic acids, polyoxyethylene alkylphenylether sulfates, polyoxyethylene styrylphenylether sulfates, and their alkali metal salts, alkaline earth metal salts, ammonium salts, and amine salts.

[Claim 2] The water-soluble granular agrochemical composition stated in Claim 1, which contains from 5 to 30 % by weight of surface-active agent 1.

[Claim 3] The water-soluble granular agrochemical composition stated in Claim 1, which contains from 1 to 10 % by weight of surface-active agent 2.

[Detailed Description of the Invention]

[0001]

* Claim and paragraph numbers correspond to those in the foreign text.

[Field of Industrial Application] The present invention pertains to a water-dispersible granular agrochemical composition having excellent disintegrability and dispersibility in water.

[0002]

[Related Art] Among agrochemical preparations, emulsions, wettable powders, flowable formulations, and the like are preparations that are diluted with water and sprayed. Because emulsions use organic solvents as the carrier, they have problems in toxicity, irritancy, inflammability, odor, and so forth. Meanwhile, because wettable powders are powder-form preparations, it is not easy to measure them, and they also pose the problem of dusting when forming them into spray solutions. Flowable formulations have solved the measuring problem and dusting problem of wettable powders by preparing them in a suspended form, but, because they are viscous liquid preparations, it is difficult to dispense them from containers, and a small amount of the formulations end up remaining inside the containers, thus posing a problem in disposing of the containers.

[0003] Accordingly, attempts have been made to form wettable powders into granules in recent years. By forming a wettable powder into granules, the dusting problem and measuring difficulty of the wettable powder can be ameliorated. In addition, granulated wettable powders, unlike flowable formulations, do not have the problem of difficulty in handling derived from high viscosity. Various proposals have been made heretofore with respect to the technique for producing

the aforesaid granulated wettable powders (the aforesaid water-dispersible granular agrochemical composition). For instance, there are a method that compounds starch and a water-soluble inorganic salt (JP-B-S53-12577), a method that compounds sugar, a naphthalenesulfonic acid-based surface-active agent, and alkali metal salt of phosphoric acid (JP-A-S57-163303), a method that combines and compounds a sulfate-based anionic surface-active agent and nonionic surface-active agent (JP-A-S59-193803), and so forth.

[0004]

[Problems that the Invention Intends to Solve] Such granulated wettable powders, however, cannot have good disintegrability and dispersibility in water when they contain agrochemical active ingredients at a high concentration, and their disintegrability and dispersibility also deteriorate during storage over time; thus, they are not necessarily practical.

[0005]

[Means for Solving the Problems] Accordingly, the present inventors conducted research extensively to solve the aforesaid problems and, as a result, found a method that can yield good disintegrability and dispersibility in water even when agrochemical active ingredients are contained at a high concentration and also that does not cause the deterioration of disintegrability and dispersibility in water during storage over time, thereby achieving the present invention.

[0006] Namely, the present invention is a water-dispersible granular agrochemical composition comprising one or more kinds of agrochemical active ingredients, surface-active agent 1 below, and surface-active agent 2 below. As surface-active ingredient 1, the present invention can use one or more kinds of surface-active agents selected from lignosulfonic acid, naphthalenesulfonic acid formalin condensate, maleic acid-diisobutylene copolymer, maleic acid-isobutylene copolymer, and their alkali metal salts, alkaline earth metal salts, ammonium salts, and amine salts. As surface-active agent 2, the present invention can use one or more kinds of surface-active agents selected from alkylnaphthalenesulfonic acids, polyoxyethylene alkylphenylether sulfates, polyoxyethylene styrylphenylether sulfates, and their alkali metal salts, alkaline earth metal salts, ammonium salts, and amine salts.

[0007] The quantity of surface-active agent 1 contained is preferably from 5 to 30 % by weight, better yet, from 10 to 20 % by weight of the agrochemical composition of the present invention. The quantity of surface-active agent 2 contained is preferably from 1 to 10 % by weight, better yet, from 2 to 5 % by weight. As the agrochemical active ingredients used in the present invention, the present invention is, though not limited specifically, applicable to those that have been used as wettable powders. The following presents typical examples thereof.

[0008] There are, as insecticides, 2-tertiary butyl-5-(4-tertiary butyl benzylthio)-4-chloropyridazine-3(2H)-one (generic name: pyridaben), 1-naphthyl-N-methylcarbamate (generic name: NAC), 3,7,9,13-tetramethyl-5,11-dioxa-2,8,14-trithia-4,7,9,12-tetraazapentadeca-3,12-diene-6,10-dione (generic name: thiodicarb), 3-methyl-1,5-bis(2,4-xylyl)-1,3,5-triazapenta-1,4-diene (generic name: amitraz), 3,6-bis(2-chlorophenyl)-1,2,4,5-tetrazine (generic name: chlofentezine), hexakis(β,β-dimethyl phenethyl)distannoxane (generic name: fenbutatin oxide), and isopropyl 4,4'-dibromobenzilate (generic name: phenysobromolate); as bactericidal agents, 2,4'-dichloro-α-(pyrimidine-5-yl)benzhydryl=alcohol (generic name: fenarimol), 8-hydroxyquinoline copper (generic name: oxine copper), 5-methyl-1,2,4-triazolo [3,4-b]benthiazole (generic name: tricyclazole), 3-(3,5-dichlorophenyl)-N-isopropyl-2,4-dioxoimidazolidine-1-carboxamide (generic name: iprodione), N-trichloromethyl thiotetrahydrophthalimide (generic name: captan), and 2,6-dichloro-4-nitroaniline (generic name: CNA); and, as herbicides, ethyl=5-(4,6-dimethoxypyrimidine-2-yl carbamoyl sulfamoyl)-1-methylpyrazole-4-carboxylate (generic name: pyrazosulfuron ethyl), methyl 5-[[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl]-3-chloro-1-methyl-1-H-pyrazole-4-carboxylate, ethyl=(RS)-2-[4-(6-chloroquinoxaline-2-yl oxy) phenoxy]propionate (generic name: quizalofop ethyl), 2-chloro-2',6'-diethyl-N-(2-propoxyethyl) acetanilide (generic name: pretilachlor), ammonium=DL-homoalanine-4-yl (methyl)phosphinate (generic name: /3

glufosinate), 5-tertiary butyl-3-(2,4-dichloro-5-isopropoxyphenyl)-1,3,4-oxadiazoline-2-one (generic name: oxadiazone), methyl-N-(3,4-dichlorophenyl)carbamate (generic name: MCC), 3-isopropyl-2,1,3-benzo-thiadiazinone-(4)-2,2-dioxide (generic name: bentazone), 2,4-dichlorophenoxyacetic acid (generic name: 2,4-D), 2-methyl-4-chlorophenoxyacetic acid (generic name: MCP), 2-methylthio-4,6-bis(isopropylamino)-s-triazine (generic name: prometryn), 2-methylthio-4-ethylamino-6-isopropylamino-s-triazine (generic name: ametryn), 2-chloro-4,6-bis(ethylamino)-s-triazine (generic name: simazine), 2-chloro-4-ethylamino-6-isopropylamino-s-triazine (generic name: atrazine), 2-methylthio-4,6-bis(isopropylamino)-s-triazine (generic name: prometryn) [sic], and the like. According to the objective, the agrochemical active ingredients can be used singly or in combination of two or more kinds.

[0009] The quantity of the contained agrochemical active ingredient is preferably from 1 to 90 % by weight, better yet, from 10 to 80 % by weight of the agrochemical composition of the present invention. The granulated wettable powder of the present invention (the aforesaid water-dispersible granular agrochemical composition) can incorporate, as necessary, a mineral powder or water-soluble powder as a bulking agent. As the mineral powder, the present invention may use diatomaceous earth, talc, clay, bentonite, calcium carbonate, and so forth. As the water-soluble powder, sugars, urea, various kinds of salts, and so forth can be used. Examples of the

sugars include lactose, fructose, glucose, and the like, and examples of the salts include alkali metal salts, ammonium salts, and the like of sulfuric acid, phosphoric acid, hydrochloric acid, nitric acid, and carbonic acid. These bulking agents may be used singly or in combination of two or more kinds.

[0010] Other auxiliary substances, such as a binding agent, grinding aid, absorbent, stabilizing agent, coloring agent, defoaming agent, and the like, may be added as necessary. The granulated wettable powder of the present invention is prepared by the following methods. Namely, according to a first preparation method, the agrochemical active ingredient, surface-active agent 1, surface-active agent 2, bulking agent, and other auxiliary substances in required quantities are combined and homogeneously mixed and subsequently ground finely. The fine grinding can be carried out with a dry mill, such as an impact grinder, ball mill, jet-o-mizer, or the like. Next, the obtained fine powder is combined with a suitable amount of water, blended, and kneaded, after which it is granulated with a granulating machine and dried, thereby obtaining the target product. The granulation can be implemented with the use of a granulator of the extrusion type, the pressure type, the fluidized-bed type, the stirring type, the tumbling type, or the like.

[0011] According to a second preparation method, to an appropriate amount of water are added required quantities of the agrochemical active ingredient, surface-active agent 1, surface-active

agent 2, bulking agent, and other auxiliary substances, and the mixture is finely ground with a wet grinding mill. The fine grinding can be implemented with the use of a wet grinding mill, such as a ball mill, sand grinder, or the like. Next, to the obtained aqueous suspension, surface-active agent 1, surface-active agent 2, the bulking agent, and other auxiliary substances are further added as necessary in appropriate quantities and blended, and this mixture is spray-dried with a spray drier, thereby obtaining the target product.

[0012] According to a third preparation method, to an appropriate amount of water are added required quantities of the agrochemical active ingredient, surface-active agent 1, surface-active agent 2, bulking agent, and other auxiliary substances, and the mixture is finely ground with a wet grinding mill. The fine grinding can be implemented with the use of a wet grinding mill, such as a ball mill, sand grinder, or the like. Next, to the obtained aqueous suspension, a bulking agent in an appropriate amount is added, concomitantly with surface-active agent 1, surface-active agent 2, and other auxiliary substances, as necessary, thereby forming a paste, which is blended and kneaded, and the thus-obtained paste is granulated with a granulator and dried, thereby obtaining the target product. The granulation can be implemented with the use of a granulator of the extrusion type, the pressure type, the fluidized-bed type, the stirring type, the tumbling type, or the like.

[0013] The grain size of the granulated wettable powder of the present invention is, though not specifically limited, preferably from 0.1 to 2 mm.

[0014]

[Working Examples] The following will explain the present invention by presenting some working examples, but the present invention is not limited to or restricted by these working examples. /4 "Parts" are all based on weight.

Working Example 1

Ninety-seven parts pyridaben (generic name) and 3 parts white carbon were finely ground with a jet-o-mizer (a product of Seishin Kigyo Co.). Seventy parts of this finely ground product, 10 parts of a mixture of a maleic acid-diisobutylene copolymer and polyoxyethylene styrylphenylether sulfate, 2.5 parts ammonium polyoxyethylene alkylphenylether sulfate, 5 parts ammonium sulfate, and 12.5 parts clay were mixed with a mixer, to which mixture was subsequently added 30 parts water and blended, and the mixture was then granulated with an extrusion granulator equipped with a screen having an opening diameter of 0.5 mm. The thus-obtained granules were dried at 50 °C and subsequently sifted through a 20 to 50-mesh sieve to regulate the grain size, thereby obtaining a granulated wettable powder of the present invention.

Working Example 2

Ninety-seven parts pyrazosulfuron ethyl (generic name) and 3 parts white carbon were finely ground with a jet-o-mizer (a product of Seishin Kigyo Co.). Seventy-five parts of this finely ground product, 10 parts of a mixture of a maleic acid-diisobutylene copolymer and polyoxyethylene styrylphenylether sulfate, 2.5 parts ammonium polyoxyethylene alkylphenylether sulfate, 5 parts ammonium sulfate, and 7.5 parts clay were mixed with a mixer, to which mixture was subsequently added 30 parts water and blended, and the mixture was then granulated with an extrusion granulator equipped with a screen having an opening diameter of 0.5 mm. The thus-obtained granules were dried at 50 °C and subsequently sifted through a 20 to 50-mesh sieve to regulate the grain size, thereby obtaining a granulated wettable powder of the present invention.

Working Example 3

Ninety-seven parts pyrazosulfuron ethyl (generic name) and 3 parts white carbon were finely ground with a jet-o-mizer (a product of Seishin Kigyo Co.). Seventy-five parts of this finely ground product, 7 parts of a formalin condensate of sodium naphthalenesulfonate, 3 parts sodium alkylnaphthalenesulfonate, and 15 parts clay were mixed with a mixer, to which mixture was subsequently added 15 parts water and blended, and the mixture was then granulated with an extrusion granulator equipped with a screen having an opening diameter of 0.5 mm. The thus-obtained granules were dried at 50 °C and subsequently sifted

through a 20 to 50-mesh sieve to regulate the grain size, thereby obtaining a granulated wettable powder of the present invention.

Working Example 4

Ninety-seven parts pyrazosulfuron ethyl (generic name) and 3 parts white carbon were finely ground with a jet-o-mizer (a product of Seishin Kigyo Co.). Seventy-five parts of this finely ground product, 7 parts of a formalin condensate of sodium naphthalenesulfonate, 2 parts sodium lignosulfonate, 2 parts sodium alkylnaphthalenesulfonate, and 14 parts clay were mixed with a mixer, to which mixture was subsequently added 15 parts water and blended, and the mixture was then granulated with an extrusion granulator equipped with a screen having an opening diameter of 0.5 mm. The thus-obtained granules were dried at 50 °C and subsequently sifted through a 20 to 50-mesh sieve to regulate the grain size, thereby obtaining a granulated wettable powder of the present invention.

Working Example 5

Ninety-seven parts quizalofop ethyl (generic name) and 3 parts white carbon were finely ground with a jet-o-mizer (a product of Seishin Kigyo Co.). Seventy-five parts of this finely ground product, 10 parts of a mixture of a maleic acid-diisobutylene copolymer and polyoxyethylene styrylphenylether sulfate, 2.5 parts ammonium polyoxyethylene alkylphenylether sulfate, 5 parts ammonium sulfate, and 7.5 parts clay were mixed with a mixer, to which mixture was subsequently added 30 parts water and blended, and the mixture was

then granulated with an extrusion granulator equipped with a screen having an opening diameter of 0.5 mm. The thus-obtained granules were dried at 50 °C and subsequently sifted through a 20 to 50-mesh sieve to regulate the grain size, thereby obtaining a granulated wettable powder of the present invention.

Working Example 6

After 55 parts pyridaben (generic name), 2 parts of a mixture of a maleic acid-diisobutylene copolymer and polyoxyethylene styrylphenylether sulfate, and 43 parts water were mixed with a homomixer, the mixture was ground with a sand grinder (a product of Imex Co.) for 90 minutes, thereby obtaining a pulverized slurry. 74.9 parts of this pulverized slurry, 13.5 parts of a mixture of a maleic acid-diisobutylene copolymer and polyoxyethylene styrylphenylether sulfate, 2.5 parts ammonium polyoxyethylene alkylphenylether sulfate, 10 parts ammonium sulfate, and 31.3 parts clay were mixed with a mixer, and the mixture was then granulated with an extrusion granulator equipped with a screen having an opening diameter of 0.5 mm. The thus-obtained granules were dried at 50 °C and subsequently sifted through a 20 to 50-mesh sieve to regulate the grain size, thereby obtaining a granulated wettable powder of the present invention.

Working Example 7

Ninety-seven parts methyl 5-[[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl]-3-chloro-1-methyl-1-H-pyrazole-4-carboxylate and 3 parts white carbon were finely ground

with a jet-o-mizer (a product of Seishin Co.). Seventy-five parts of this finely ground product, 7 parts of a formalin condensate of sodium naphthalenesulfonate, 2 parts sodium lignosulfonate, 2 parts sodium alkylnaphthalenesulfonate, and 14 parts clay were mixed with a mixer, to which mixture was subsequently added 30 parts water and blended, and the mixture was then granulated with an extrusion granulator equipped with a screen having an opening diameter of 0.5 mm. The thus-obtained granules were dried at 50 °C and subsequently sifted through a 20 to 50-mesh sieve to regulate the grain size, thereby obtaining a granulated wettable powder of the present invention.

Comparative Example 1

Ninety-seven parts pyridaben (generic name) and 3 parts white carbon were finely ground with a jet-o-mizer (a product of Seishin Kigyo Co.). Seventy parts of this finely ground product, 10 parts of a maleic acid-diisobutylene copolymer, 5 parts ammonium sulfate, and 15 parts clay were mixed with a mixer, to which mixture was subsequently added 30 parts water and blended, and the mixture was then granulated with an extrusion granulator equipped with a screen having an opening diameter of 0.5 mm. The thus-obtained granules were dried at 50 °C and subsequently sifted through a 20 to 50-mesh sieve to regulate the grain size, thereby obtaining a granulated wettable powder of the present invention. /5

Comparative Example 2

Ninety-seven parts pyrazosulfuron ethyl (generic name) and 3 parts white carbon were finely ground with a jet-o-mizer (a product of Seishin Kigyo Co.). Seventy-five parts of this finely ground product, 10 parts of a maleic acid-diisobutylene copolymer, 5 parts ammonium sulfate, and 10 parts clay are mixed with a mixer, to which mixture was subsequently added 30 parts water and blended, and the mixture was then granulated with an extrusion granulator equipped with a screen having an opening diameter of 0.5 mm. The thus-obtained granules were dried at 50 °C and subsequently sifted through a 20 to 50-mesh sieve to regulate the grain size, thereby obtaining a granulated wettable powder of the present invention.

Comparative Example 3

Ninety-seven parts pyrazosulfuron ethyl (generic name) and 3 parts white carbon were finely ground with a jet-o-mizer (a product of Seishin Kigyo Co.). Seventy-five parts of this finely ground product, 7 parts of a formalin condensate of sodium naphthalenesulfonate, and 18 parts clay were mixed with a mixer, to which mixture was subsequently added 15 parts water and blended, and the mixture was then granulated with an extrusion granulator equipped with a screen having an opening diameter of 0.5 mm. The thus-obtained granules were dried at 50 °C and subsequently sifted through a 20 to 50-mesh sieve to regulate the grain size, thereby obtaining a granulated wettable powder of the present invention.

Comparative Example 4

Ninety-seven parts quizalofop ethyl (generic name) and 3 parts white carbon were finely ground with a jet-o-mizer (a product of Seishin Kigyo Co.). Seventy-five parts of this finely ground product, 7 parts of ammonium polyoxyethylene alkylphenylether sulfate, 5 parts ammonium sulfate, and 13 parts clay were mixed with a mixer, to which mixture was subsequently added 30 parts water and blended, and the mixture was then granulated with an extrusion granulator equipped with a screen having an opening diameter of 0.5 mm. The thus-obtained granules were dried at 50 °C and subsequently sifted through a 20 to 50-mesh sieve to regulate the grain size, thereby obtaining a granulated wettable powder of the present invention.

Comparative Example 5

Ninety-seven parts methyl 5-[[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl]-3-chloro-1-methyl-1-H-pyrazole-4-carboxylate and 3 parts white carbon were finely ground with a jet-o-mizer (a product of Seishin Co.). Seventy-five parts of this finely ground product, 7 parts of sodium alkylnaphthalenesulfonate, and 18 parts clay were mixed with a mixer, to which mixture was subsequently added 30 parts water and blended, and the mixture was then granulated with an extrusion granulator equipped with a screen having an opening diameter of 0.5 mm. The thus-obtained granules were dried at 50 °C and subsequently sifted through a 20 to 50-mesh sieve to regulate the grain size, thereby obtaining a granulated wettable powder of the present invention.

[0015] Next, the preparations obtained in the working examples and comparative examples were tested for disintegrability in water and suspension stability.

Test Example 1: In-water disintegrability test

1.0 g of a test sample was precisely measured. The measured sample was charged into a 100 ml-capacity stoppered graduated cylinder that contained 100 ml of 20 °C, 3-degree hard water. After the cylinder was kept still for 20 seconds, the measuring cylinder was inverted at a rate of once every 2 seconds, and the number of inversions that were made until the granulated wettable powder was completely disintegrated was taken as the disintegrability of the sample in water. A smaller number of inversions is considered to be better. The results are shown in Table 1.

Test Example 2: Suspension stability test

1.0 g of a test sample was weighed precisely and placed in a 100 ml-capacity beaker, to which 50 ml of 20 °C, 3-degree hard water was added, and the mixture was thoroughly blended and dispersed. This was transferred to a 250 ml-capacity plugged cylinder, to which was further added 20 °C, 3-degree hard water to set the total quantity to 250 ml. After this cylinder was kept still for 15 minutes, it was vigorously shaken 30 times in 1 minute and then kept still for 5 minutes. Next, a 25 ml-capacity volumetric pipette was put in the liquid, and its tip was kept at the center of the liquid to gently sample 25 ml of the sample liquid. The dry constant mass (residue on

drying) in the sample liquid was measured, and the suspension rate was found from the expression below.

[0016]

Suspension rate (%) = [(Bx10)/A] x 100

A: the weight of the sample that was weighed precisely at the start;

B: the weight of the residue in the sample liquid on drying.

A larger suspension rate is considered to be better. The results are shown in Table 1.

TABLE 1

(a) 水中崩壊性 (回)	(b) 懸濁率 (%)
実施例1(c)	5	94
実施例2(c)	3	98
実施例3(c)	3	97
実施例4(c)	3	98
実施例5(c)	3	93
実施例6(c)	5	92
実施例7(c)	3	98
比較例1(d)	20以上(e)	25
比較例2(d)	20以上(e)	31
比較例3(d)	20以上(e)	43
比較例4(d)	20以上(e)	58
比較例5(d)	20以上(e)	62

Key: a) disintegrability in water (times); b) suspension rate; c) working example; d) comparative example; e) 20 or more

[0017]

[Effects of the Invention] The agrochemical compositions of the present invention have excellent disintegrability and dispersibility in water.